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EFFECT OF OXYGEN ON THE TEAR STRENGTH OF ELASTOMERS

by

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Rates of tearing under steady and intermittent loads have been measured for some representative elastomers. The rates depended strongly upon the load and also upon the environment. Tearing was much slower for SBR and BR vulcanizates in vacuo, especially at low loads, by a factor of up to 10X. For NR the effect was quite small, by a factor of 2X at the most. Experiments were also carried out in the presence of thiophenol vapor, a radical trap. For NR, the effect

was found only at high loads, when tearing was much slower, by a factor of about 10X. The improvement persisted after thiophenol was removed; it is attributed chemical modification of the vulcanizate. On the other hand, tearing of SBR was accelerated at all loads by thiophenol vapor. to the same degree as in air. Much smaller effects were found for a peroxide vulcanizate of SBR. It is concluded that the reactions of sulfur crosslinks with radical acceptors, including oxygen, lead to an increased rate of tearing in SBR. For BR and NR vulcanizates the situation is less clear, because of the smaller effect of oxygen in the first case and of thiophenol in the second.

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1. Introduction

Many measurements have been made of the strength of simple vulcanizates of common elastomers, both under steady and intermittently applied loads. In both cases, the strength is governed by dissipative processes, particularly those arising from internal molecular motions and from stress-induced crystallization (1). Thus, the intrinsic strength of the molecular network is only observable under non-dissipative conditions, at high temperatures and extremely low rates of tearing (2-4). Under these circumstances the strength of rubber networks is in reasonably good agreement with molecular calculations based on the dissociation energy of main-chain bonds in the molecular strands comprising the network (5).

Relatively few measurements have been made under different atmospheres. In vacuo, the mechanical fatigue life of an NR vulcanizate was found to be increased by a factor of about 10X (6). Similarly, the rate of tear propagation under intermittently applied loads was found to be reduced by a factor of 3-4 in vacuo (7) and for a carbon-black-filled NR vulcanizate in nitrogen by a similar but somewhat smaller factor of 1.8 (8). And the tensile strength of an SBR vulcanizate at high temperature was found to be increased in vacuo, by a surprisingly large amount, about 45 percent on average (9). Thus, there is much evidence for an effect of the environment on the strength of elastomers, the strength being significantly lowered in air. But the reason for this effect is obscure. Is it due to oxidation of highly-stretched molecules before rupture? Or are polar molecules

weaker in the presence of air (oxygen)? Do additional fractures take place in the presence of air (oxygen) that add to the amount of tearing? Another possibility is that broken molecules recombine to some extent when tension is removed in the absence of oxygen, but not in its presence, because oxygen is a powerful radical trap and could prevent polymer radicals from combining with other molecules.

In an attempt to clarify the magnitude and origin of environmental effects, we have carried out an experimental study of the rates of tearing of simple vulcanizates of three elastomers; NR, SBR and BR; in air, in vacuo, and in the presence of thiophenol vapor, a recognized radical acceptor. The results are reported here.

2. Experimental details

Mix formulations and vulcanization conditions are given in Table 1. They produced soft elastic materials with a tensile (Young) modulus between 1.5 and 2.2 MPa. Rates of crack propagation were determined using long narrow specimens bonded between two parallel brass strips as shown in Figure 1, pulled apart in the perpendicular direction. Resembling pure shear specimens, they allowed the measurement of crack growth over long distances under constant conditions.

An initial cut, about 30 mm long, was made in one end of the specimen and the tip was sharpened with a lubricated razor blade. The specimen was then strained to a given degree and either held in this state (steady tearing) or relaxed completely and stretched again to the same degree repeatedly (intermittent tearing). Growth of the cut was

measured with a travelling microscope. After a small distance of rapid growth, tearing took place over long distances at a constant rate that depended strongly upon the applied strain.

From measured relations for uncut specimens between tensile load F and displacement δ of one bonded brass strip with respect to the other, the amount of elastic energy W' stored per unit length of the specimen was obtained as a function of the average strain ϵ, where ϵ is given by δ/h_o and h_o is the original separation of the brass strips (30 mm). The results for three vulcanizates are shown in Figure 2. From these relations the energy G released per unit area torn through by growth of a crack along the center of the specimen was obtained:

$$G = W'/t_o \tag{1}$$

where t_o is the thickness of the central part of the specimen (about 0.75 mm) (10). The quantity G is termed "tearing energy" hereafter.

Rates of crack growth were measured at various applied strains ϵ. They are discussed below in terms of corresponding values of the tear energy G, for ready comparison with other work.

A specimen was subjected either to a steady strain, applied continuously, or to an intermittently-applied strain at a frequency of 1.8 Hz. In the latter case, care was taken to ensure that the specimen was fully relaxed during each strain cycle. To compare the two results the effective time under load was computed in the intermittent case by estimating the fraction of time that the specimen was subjected to a strain of at least 90 percent of the maximum value, i.e., with strain energy of at least 80 percent of the maximum amount. It was concluded

that about 18 percent of the total time was spent in this way.

The test specimen was enclosed in a sealed chamber as shown schematically in Figure 3. Strains were applied to it through a flexible seal, consisting of a long lubricated rubber tube fastened at one end to the chamber and at the other end to a long steel rod that passed down the center of the tube and was used to apply deflections to the specimen (6). Because of its length, about 500 mm, the rubber tube seal only experienced small tensile strains and did not undergo fatigue cracking during the course of the experiments.

The test chamber had a volume of about 1.5 L. It could be evacuated by an oil pump during an experiment, taking about 15 min to reach 1×10^{-3} mm of Hg. Pumping continued thereafter to maintain this level of vacuum. The crack thus grew first in air and then _in vacuo_. Air was readmitted if desired, and re-evacuated, to examine the reproducibility of crack growth rates in different atmospheres. Provision was also made to admit other materials. A reservoir containing about 0.1 ml of liquid thiophenol was opened after evacuating the chamber, creating a low-concentration atmosphere, less than 10^{-4} g/L, of thiophenol vapor within the chamber.

All tests were carried out at room temperature.

2. Experimental results and discussion

Crack growth under intermittent stressing

The crack length c is plotted in Figure 4 against the number n of strain cycles for an NR vulcanizate subjected to a maximum strain ϵ of 16.3 percent, corresponding to a tear energy G of 700 J/m^2. A well-defined rate of crack growth is evident, given by the slope $\partial c/\partial n$, and it is somewhat lower in vacuo than in air. But the effect is rather small. For an SBR vulcanizate, on the other hand, the effect was considerably larger, Figure 5. In this case the rate of crack growth in air was about five times larger than in vacuo. Similar results were obtained for NR by Lindley and Thomas (7), who measured crack growth rates in air 3 to 4 times greater than in vacuo, and by Young (8), who found crack growth to be about twice as fast as in vacuo for a carbon-black-filled NR vulcanizate. No other measurements of crack growth in vacuo are known to the present authors. Results for several compounds of NR, SBR and BR are presented later.

Rates of crack growth in air for sulfur vulcanizates of NR and SBR are plotted against tear energy G in Figure 6, using logarithmic scales for both axes. In this representation the results lie on straight lines approximately, at least over a large range of values for G, with rather different slopes for the two materials. Thus, the rates of crack growth are given by

$$\partial c/\partial n - AG^{\alpha} \qquad (2)$$

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where $\underline{A}$ is a crack growth constant and the index $\underline{\alpha}$ takes values of about 2 for NR and about 4 for SBR. These differences are attributed to different degrees of mechanical hysteresis in the two materials, greater in NR than in SBR because of stress-induced crystallization in NR (1). A lower limit for $\underline{\alpha}$ of 2 has been deduced for completely dissipative materials, because the stress distribution around a sharp crack leads to predicted growth steps that depend upon $\underline{G}$ in this way (11). For relatively elastic materials, on the other hand, when the stress distribution propagates with the growing crack, the value of $\underline{\alpha}$ is found to be much larger (12), rising towards infinity for a perfectly elastic solid. Results for BR, a highly-elastic material, were found to be in accord with a value of 5-6 (see Figure 14, later).

Results obtained by Lake and Lindley (12) for unfilled NR and SBR materials of similar composition are represented by the broken lines in Figure 6. In view of possible differences in mix formulations and methods of measurement, the agreement between the present results and those of Lake and Lindley is considered to be reasonably good. However, at large values of tearing energy the rate of crack growth in NR suddenly increased, by a factor of about 100. The growth step increased from about 100 nm per load application to about 10 μm, at a critical value of $\underline{G}$ of about 2,200 J/m^2, Figure 6. A similar phenomenon was observed by Lake and Lindley (12) but at a considerably larger value of $\underline{G}$, about 10,000 J/m^2.

No correspondingly abrupt change in $\partial c / \partial n$ was noticed with SBR or BR materials. The effect may therefore be due to a transition in the

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length of the growth step per cycle, relative to the size of the partially-crystalline zone set up in NR by high stresses at the crack tip (13). As shown later, small changes in NR materials caused dramatic differ es in the critical tearing energy at which this transition occurred.

We now discuss the effects of the environment on crack growth in NR, a stress-crystallizing material, separately from the effects on crack growth in SBR and BR vulcanizates.

Crack growth in NR

It has already been seen that growth rates in vacuo were not much lower than in air (Figure 7). Similarly, when an antioxidant (N-isopropyl-N'-phenyl-paraphenylene diamine, Santoflex IP) was included in the mix formulation the growth rates in air were only slightly reduced, Figure 8. However, the critical tear energy, at which a sudden increase in crack growth rate occurred, was much smaller than for the unprotected material. It is possible that the antioxidant affected the type or degree of crosslinking in some way, but further work would be necessary to clarify this effect

When thiophenol vapor was admitted to the evacuated test chamber, the rates of crack growth for a wide range of tearing energy were hardly affected, Figure 9. But the critical tearing energy was dramatically increased in this case, as shown in Figure 9. Fracture surfaces in thiophenol-treated materials were noticed to be much more irregular than in untreated NR, showing some secondary cracks and rougher torn surfaces.

The reinforcing effect of thiophenol persisted even after the vapor was removed, and the test continued in vacuo. It was also evident in samples that were tested in air after exposure to thiophenol vapor. Figure 9. Measurements of load-deflection relations for the treated samples showed that there had been no significant change in the elastic properties of the material. Apparently, treatment with thiophenol vapor for several hours at room temperature had caused a slight but significant chemical change in the vulcanizate that greatly reduced the rate of crack growth at high stresses without affecting the elastic behavior. It seems possible that a slight chemical modification of the vulcanizate had occurred and altered its propensity to crystallize at high stresses.

Cunneen et al demonstrated that some cis-trans isomerization occurs when NR is treated with thiophenol in vacuo (14). If this is the case in our experiments it is surprising that the resistance to cracking is increased, because structural irregularities would be expected to hinder crystallization, not enhance it. Perhaps too rapid or too great a degree of crystallinity is not as advantageous in preventing crack growth as a smaller amount.

It was not found possible to carry out crack growth experiments under intermittent loading for peroxide (carbon-carbon crosslinked) vulcanizates of NR. Crack growth steps were found to be erratic, changing abruptly from distances too small to measure to distances of many mm in experiments carried out under apparently the same conditions. The reason for this erratic tearing behavior is not known. On the

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other hand, cracks grew in peroxide vulcanizates of SBR and BR at well-defined rates under intermittent loading. Experimental values for these materials are reported later and compared with those for sulfur vulcanizates.

No crack growth occurs in NR materials when a steady load is applied (provided that the load is not so large that catastrophic tearing results, at values of fracture energy of about 5 kJ/m^2 or more), because crystallinity at the crack tip presents a permanent barrier (1). But cracks grow in non-crystallizing materials, like SBR and BR, under a steady load, at rates depending upon the applied load. Rates of steady tearing for these materials are reported below and compared with rates of crack growth under intermittent loading.

Crack growth in SBR under intermittent loads

Crack growth rates for sulfur vulcanizates of SBR in air, in vacuo, and in thiophenol vapor are plotted against tearing energy G in Figures 10 and 11. Rates in vacuo were always lower than in air by factors of between 3 and 8. When thiophenol vapor was introduced into the test chamber, the rate of crack growth increased immediately to about the same rate as in air. On removing the thiophenol vapor, the rate reverted to its original low value in vacuo. For SBR, therefore, exposure to thiophenol had no permanent effect.

The pronounced effect of air (oxygen) and thiophenol on the rate of crack growth suggests that free-radical reactions can contribute to the size of the growth step, in addition to the fundamental component due to mechanical rupture of highly stressed chains. One possible mechanism

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for the accelerating effect of oxygen and other radical acceptors like thiophenol on the rate of tearing is that they react with some of the broken molecules and prevent them from recombining or linking with other molecules when the specimen is relaxed. In effect, they prevent some degree of healing from taking place. This hypothesis was examined by studying crack growth under steady loads, when healing cannot take place because the specimen is never relaxed.

Crack growth in SBR under steady loads

As shown in Figure 12, rates of tearing under steady loads were found to depend even more strongly upon the tear energy $\underline{G}$ than for intermittent loading, in accord with a higher value for the index $\underline{\alpha}$ of about 6.5 in the empirical relation

$$\partial c/\partial t = A'G^{\alpha} \tag{3}$$

where $\underline{A}'$ is a growth constant for steady tearing. Again, rates of tearing in vacuo were much smaller than in air, and they returned to the values in air, or even exceeded them, when thiophenol vapor was introduced. For example, at a fracture energy level of 1 kJ/m^2, the rate of tear propagation in air was 270 nm/s, in vacuo it was reduced to 9 nm/s, and in the presence of thiophenol vapor it increased to 300 nm/s. Corresponding results at a fracture energy level of 1.46 kJ/m^2 were 3.5 $\mu m/s$, 0.25 $\mu m/s$ and 24 $\mu m/s$. These observations clearly demonstrate that the "healing" hypothesis is incorrect. Crack growth is accelerated by radical acceptors both under steady and intermittent

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loads. It must be concluded that the material is weakened by the presence of a radical acceptor and tears more easily.

In order to compare rates of tearing under steady and intermittent loads, the effective time under load was calculated for intermittent loading by taking one strain cycle to be equivalent to a time under maximum load of 0.1 s, i.e., 18 per cent of the period of the cyclic motion. Measured rates of crack growth $\partial c/\partial n$ were replotted on this basis as $\partial c/\partial t$. As can be seen in Figure 12, the rates under intermittent loading were much higher, by three orders of magnitude, than the directly-measured rates of steady tearing. Nevertheless, the effects of applied load, and of radical acceptors, were qualitatively the same in the two types of experiment.

Crack growth in a peroxide vulcanizate of SBR was about 30 times faster than for the sulfur vulcanizate, Figure 13, although it followed the same strong dependence on tear energy, with a value for α of about 6. Greater resistance to tearing in sulfur vulcanizates has been observed before and attributed to labile sulfur bonds, breaking and reforming to release internal stresses (14). Sulfur vulcanizates are more dissipative as well, probably for the same reason.

For the peroxide-vulcanized material, however, the results in air and in vacuo were not very different, being slower in vacuo by only a factor of 2 at most. Thus, the pronounced effect of a radical acceptor on tear strength was largely absent. We must conclude that the effect of the presence of a radical acceptor in the environment is associated mainly with sulfur crosslinks, rather than carbon-carbon bonds.

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Presumably, oxygen and thiophenol react with $RS_n\cdot$ rather than with $R\cdot$ radicals in the present experiments.

Polybutadiene

Rates of crack growth for a sulfur vulcanizate of BR are plotted in Figure 14 against the tear energy $\underline{G}$. They were much higher than for SBR, reflecting the weak tear resistance of BR, associated with its highly-elastic, non-dissipative nature. Again, the rates under intermittent loading were generally much higher than those under steady loading, by a factor of up to 100X. But at high tear energies the rates became similar. There was little if any enhancement of tearing by intermittent loading at tear energies of 500 J/m^2 or greater.

Similarly, when air was removed from the test chamber the rate of tearing was reduced by a factor of about 10 at low tear energies, as for a sulfur vulcanizate of SBR, but at high tear energies the effect diminished, Figure 14. Consequently, the value of the index $\underline{\alpha}$ in Equations 1 and 2 was higher in vacuo than in air, about 6 instead of 5, and still higher under steady tearing, about 10, than under intermittent tearing.

A limited number of experiments were carried out with thiophenol vapor. It appeared to have little effect, the rate of tearing remaining at in vacuo levels. For example, at a fracture energy level of 0.22 kJ/m^2 the rate of tearing in air was 2.1 $\mu m/s$, in vacuo it was reduced to 0.56 $\mu m/s$, and in thiophenol vapor it was even lower, only 2.7 $\mu m/s$. For a higher fracture energy, of 0.36 kJ/m^2, the corresponding tear rates were 46 $\mu m/s$, 17 $\mu m/s$, and 23 $\mu m/s$. Thus,

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although BR showed the same effect of air (oxygen) in accelerating both steady and intermittent tearing, as for SBR, thiophenol vapor did not produce a similar effect. Possibly radicals formed by bond rupture in the polybutadiene vulcanizate were less able to react with thiophenol.

4. Conclusions

1. Environmental effects on the rate of crack growth in NR were rather small. Rates in vacuo were about one-half of those in air. Thiophenol vapor had little effect at low loads. Apparently, stress-induced crystallinity is the dominant factor governing crack growth in NR, rather than chemical reactivity.

2. Treatment of NR with thiophenol vapor in vacuo caused a substantial increase in the critical tear energy at which crack growth rates increased abruptly. As a result, the material had much better tear resistance at high loads. This improvement persisted after removing thiophenol. It is attributed to a slight chemical modification of the material by treatment with thiophenol, changing its propensity to crystallize on stretching.

3. The test environment plays an important role in both steady and intermittent tearing of sulfur vulcanizates of SBR and BR. In vacuo, rates of tear were about 8 times lower than in air in both experiments and for both materials. Thiophenol accelerated tearing to the same degree as air for SBR, but had little if any effect on tearing of BR.

4. Much smaller effects were observed for a peroxide vulcanizate of SBR. It is therefore proposed that sulfur bonds are weakened by the

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presence of a radical acceptor, and tearing is accelerated as a result.

5.   Environmental effects were generally less significant at high loads and high rates of tear.   Apparently the reactions that accelerate tearing became less important when the growth step was large or the rate of tear was high.

Acknowledgements

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1. Crack growth test piece.

2. Elastic strain energy W' per unit length of specimen $\underline{vs}$ average strain $\underline{\epsilon}$.

3. Test apparatus.

4. Crack growth $\underline{c}$ $\underline{vs}$ number $\underline{n}$ of strain cycles for an NR vulcanizate. $\underline{\epsilon}_{max} = 0.163$, $\underline{G}_{max} = 700$ J/m^2. Filled symbols, in air; open symbols, $\underline{in}$ $\underline{vacuo}$.

5. Crack growth $\underline{c}$ $\underline{vs}$ number $\underline{n}$ of strain cycles for an SBR vulcanizate. $\underline{\epsilon}_{max} = 0.09$, $\underline{G}_{max} = 275$ J/m^2. Filled symbols, in air; open symbols, $\underline{in}$ $\underline{vacuo}$.

6. Crack growth per strain cycle $\underline{vs}$ tear energy $\underline{G}$. Filled symbols, NR; open symbols, SBR. Broken lines represent the results of Lake and Lindley (12).

7. Crack growth per strain cycle $\underline{vs}$ tear energy $\underline{G}$ for NR in air (filled symbols) and $\underline{in}$ $\underline{vacuo}$ (open symbols).

8. Crack growth per strain cycle $\underline{vs}$ tear energy $\underline{G}$ for NR containing an antioxidant NR-A (open symbols) and unprotected (filled symbols).

9. Crack growth per strain cycle $\underline{vs}$ tear energy $\underline{G}$ for NR. In air, ● ; in thiophenol vapor, △ ; after treatment with thiophenol vapor for two hours, ○ .

10. Crack growth per strain cycle $\underline{vs}$ tear energy $\underline{G}$ for SBR. In air, ● ; $\underline{in}$ $\underline{vacuo}$, □ ; in thiophenol vapor, △ .

11. Crack growth per strain cycle vs tear energy G for an SBR vulcanizate containing an antioxidant. In air, ● ; in vacuo, ▢ ; in thiophenol vapor, △ .

12. Crack growth rates for SBR vs tear energy G. Filled symbols, intermittent tearing; open symbols, steady tearing. Circles, in air; squares, in vacuo.
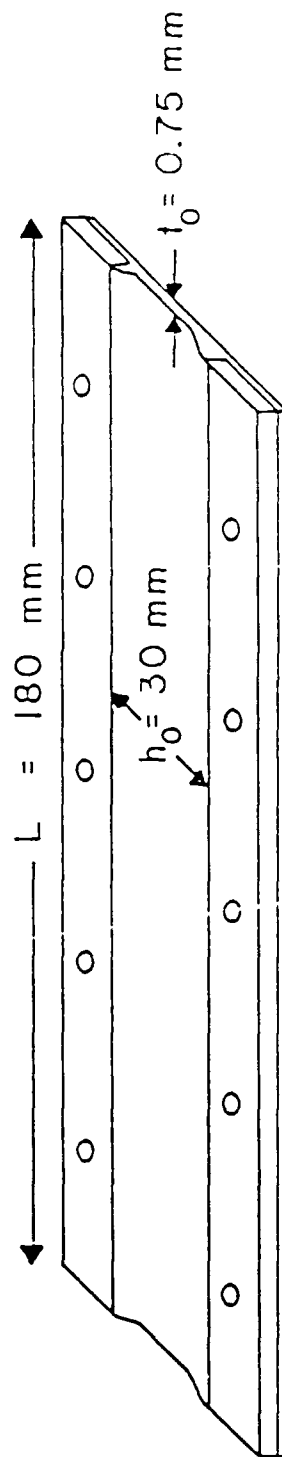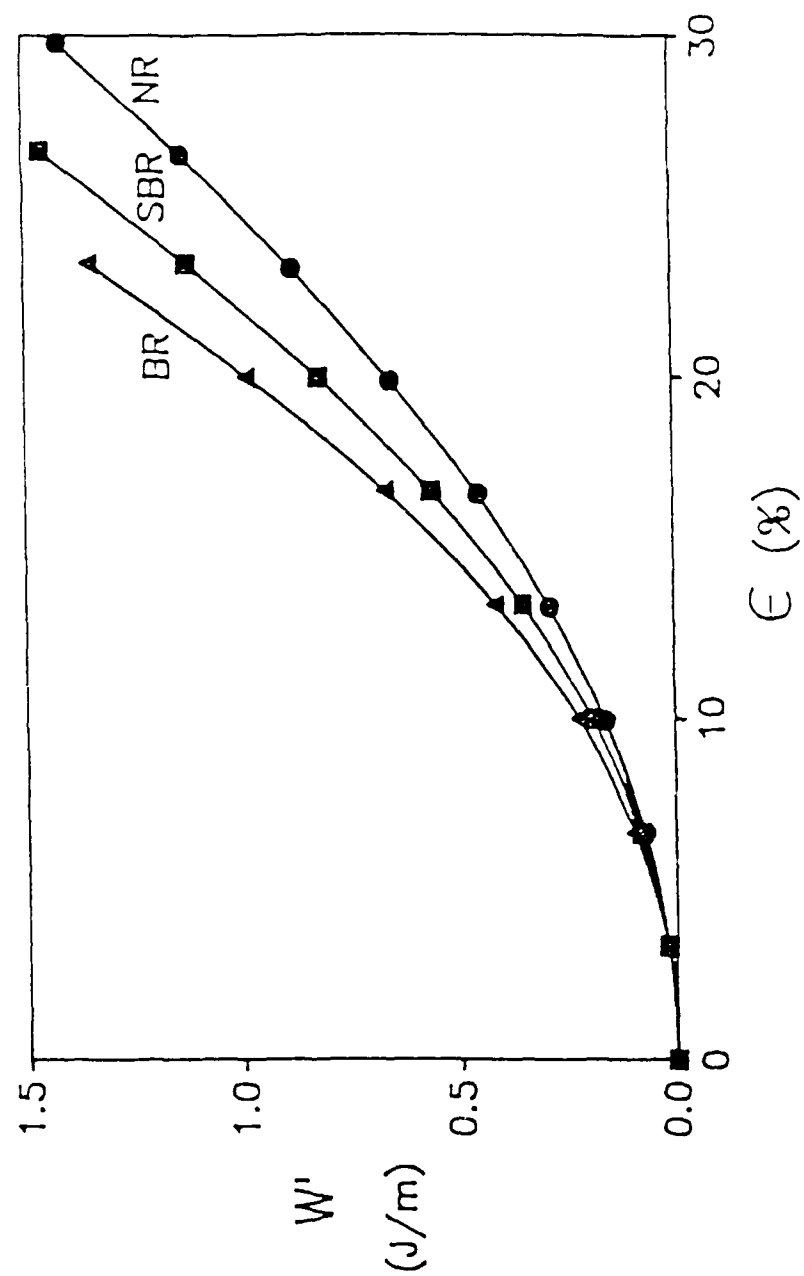
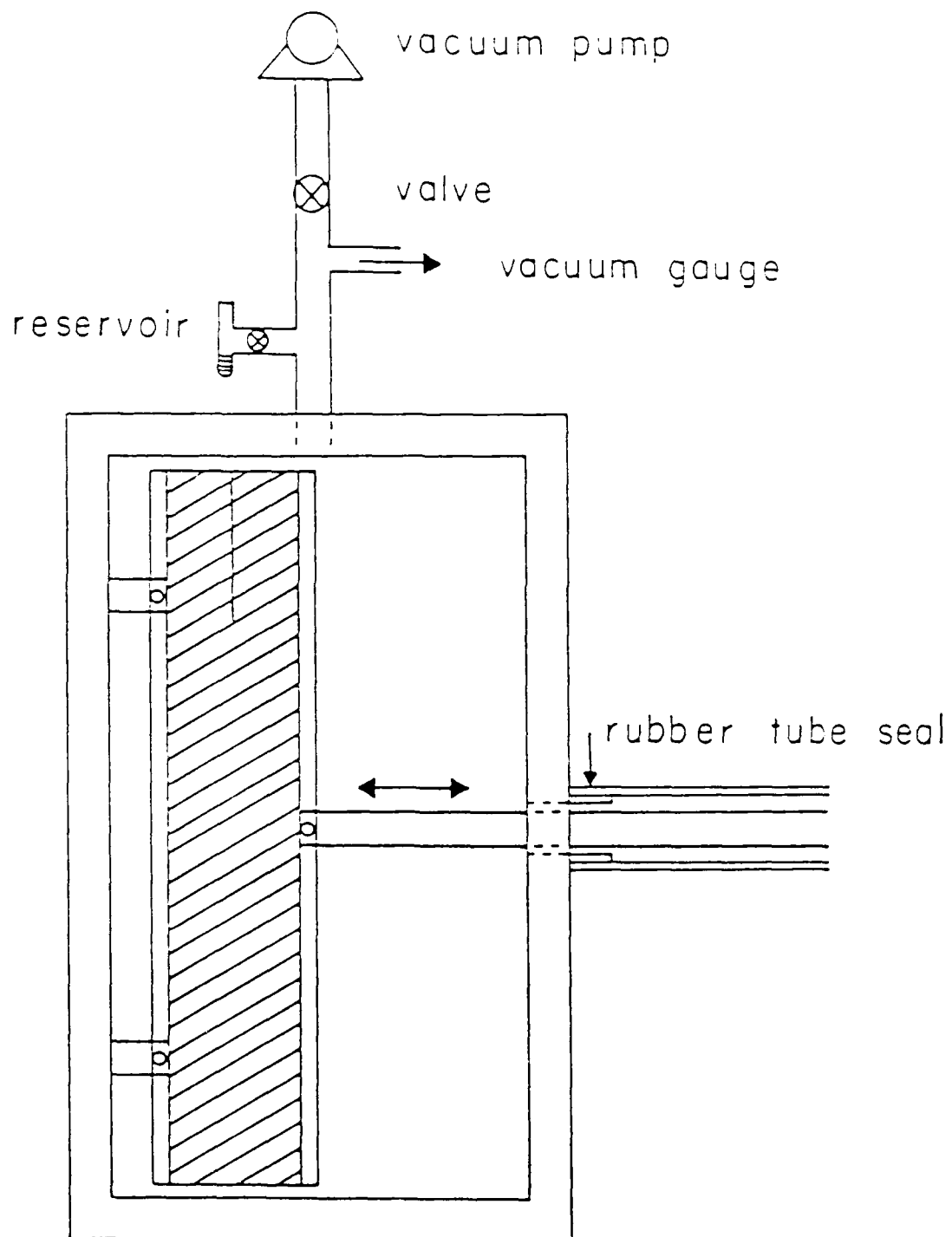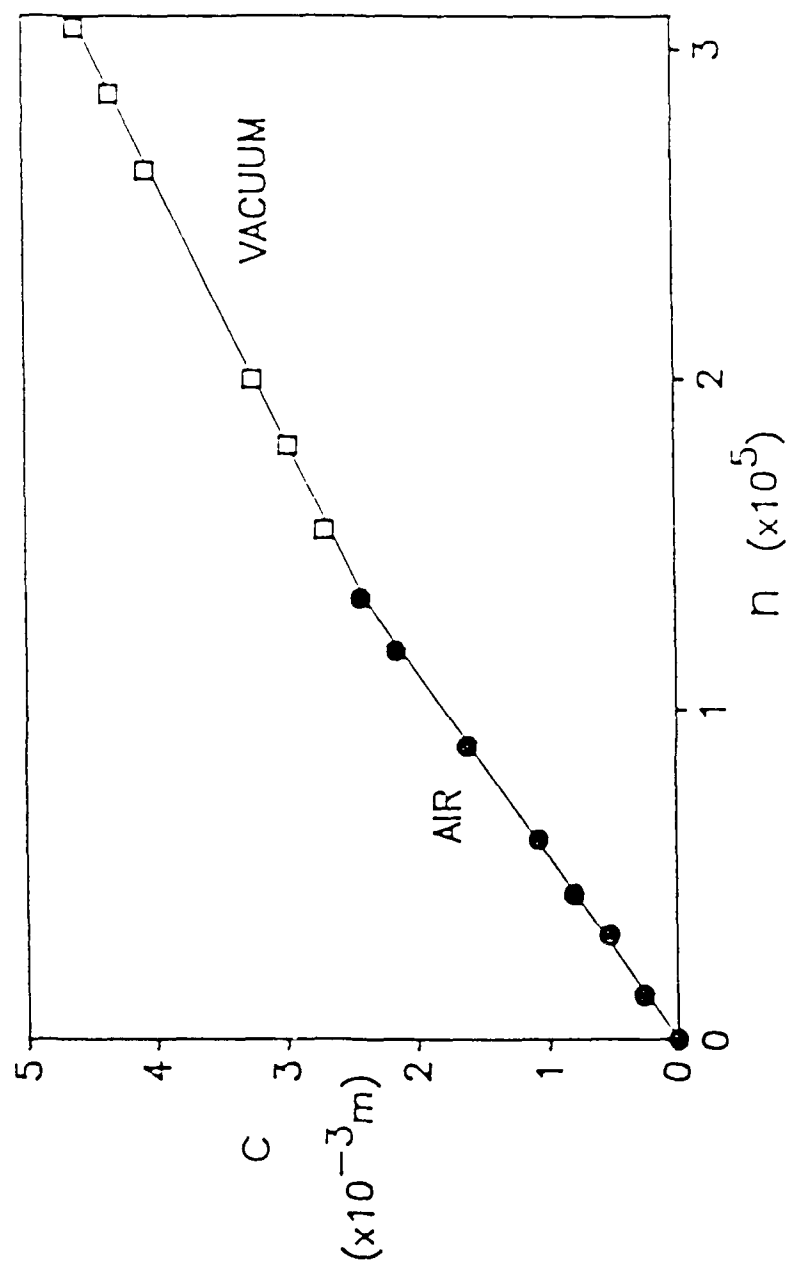13. Rates of steady tearing for SBR vulcanizates vs tear energy G. Filled symbols, peroxide-cured samples; open symbols, sulfur-cured samples. Circles, in air; squares, in vacuo.

14. Crack growth rates for BR vs tear energy G. Filled symbols, intermittent tearing; open symbols, steady tearing. Circles, in air; squares, in vacuo.

FIGURE 1

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FIGURE 2

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vacuum pump

valve

vacuum gauge

reservoir

rubber tube seal

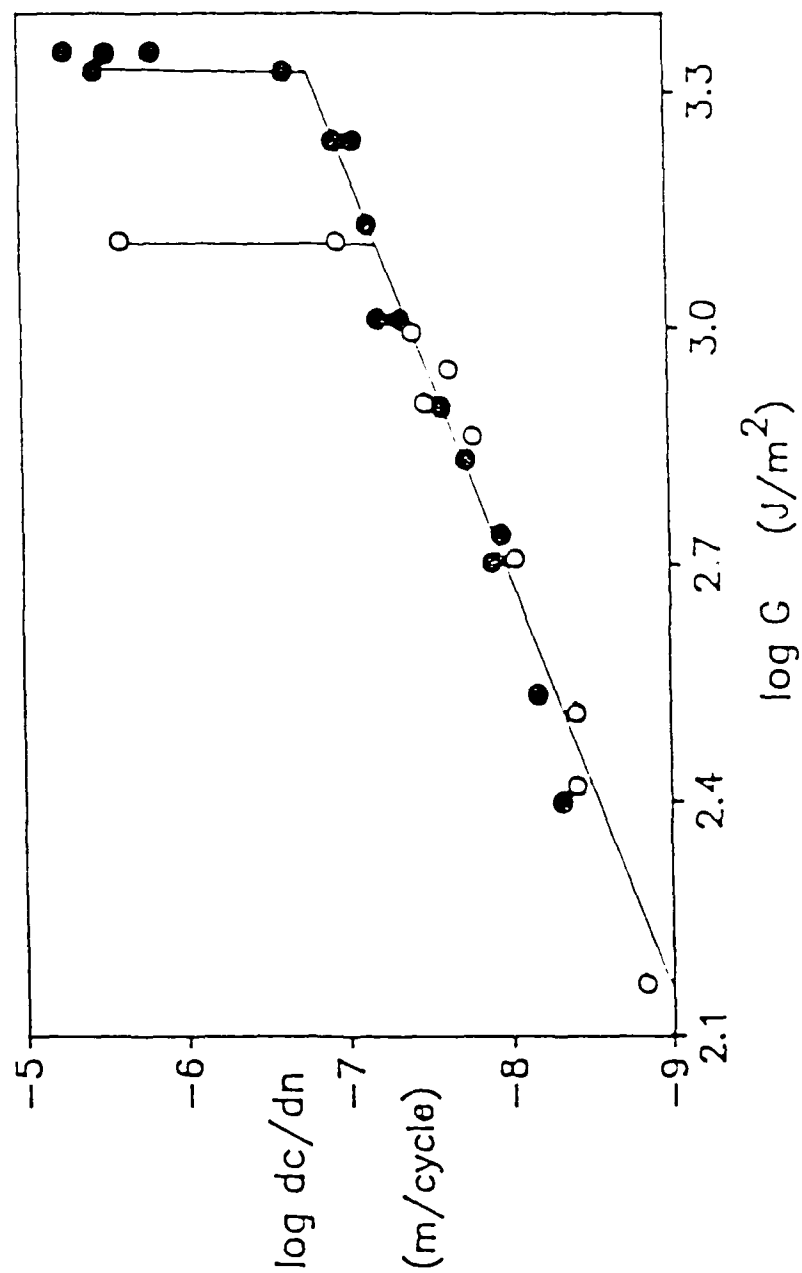FIGURE 3

FIGURE 4

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FIGURE 5
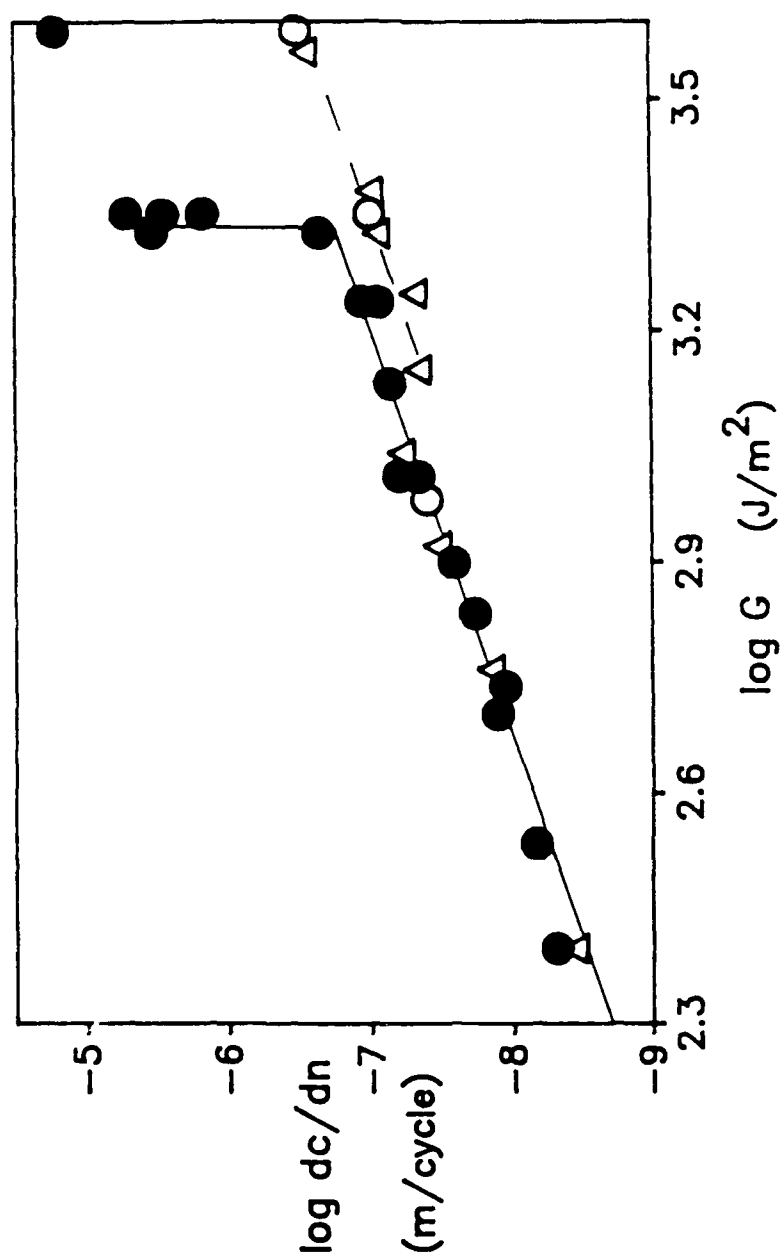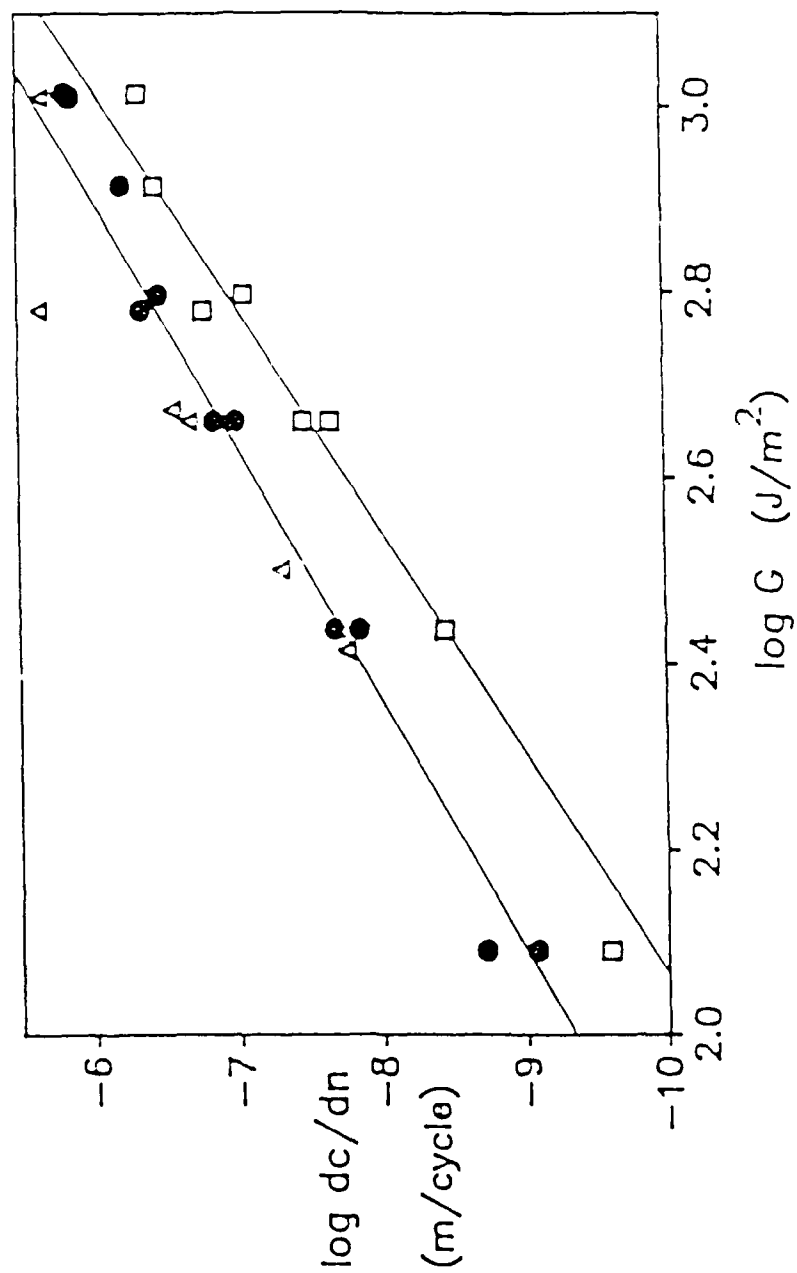
FIGURE 6

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FIGURE 7

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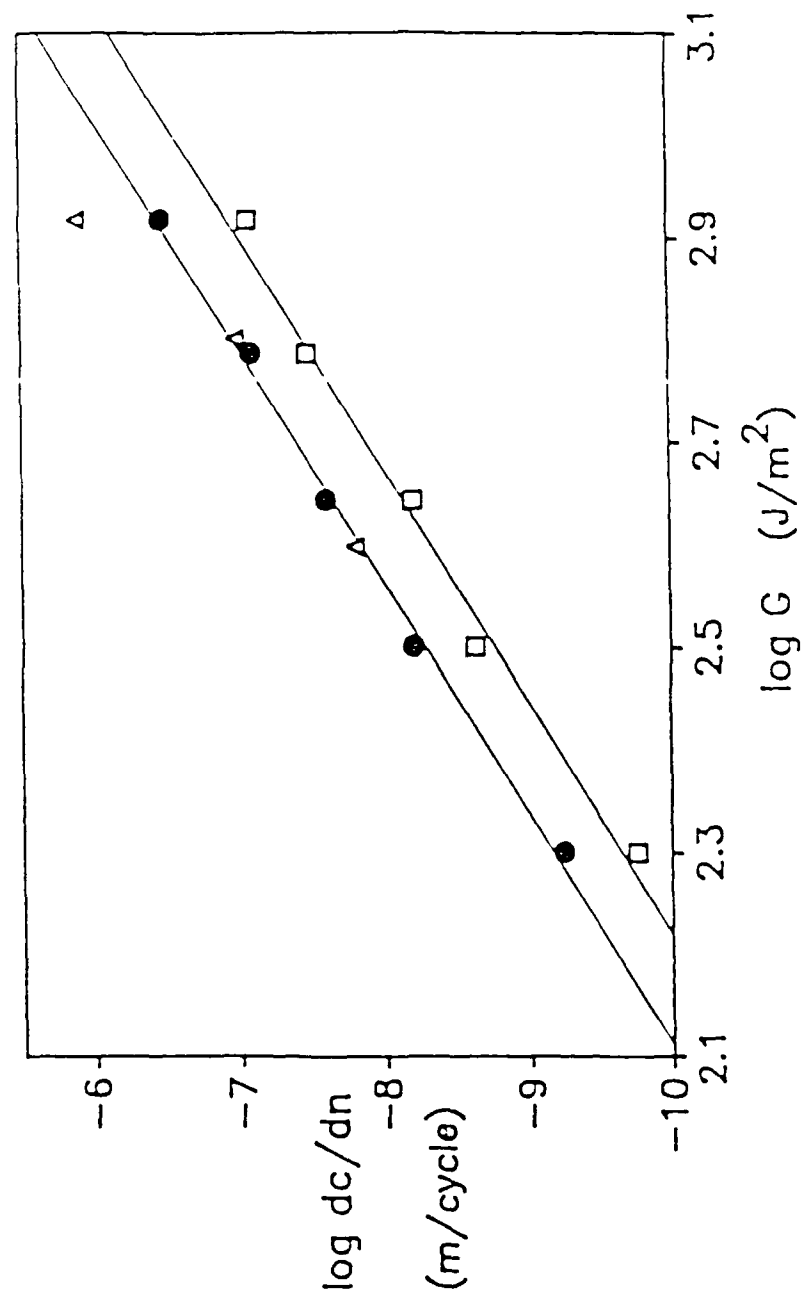FIGURE 8

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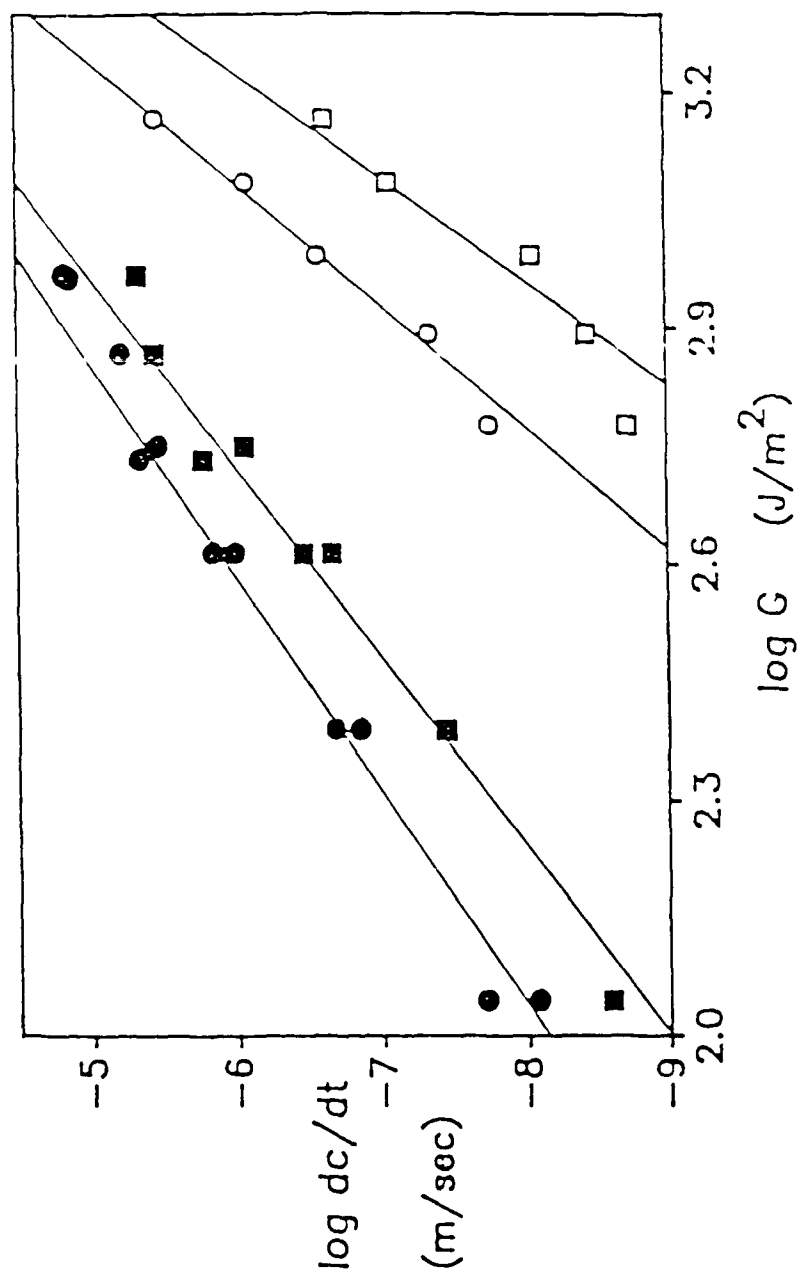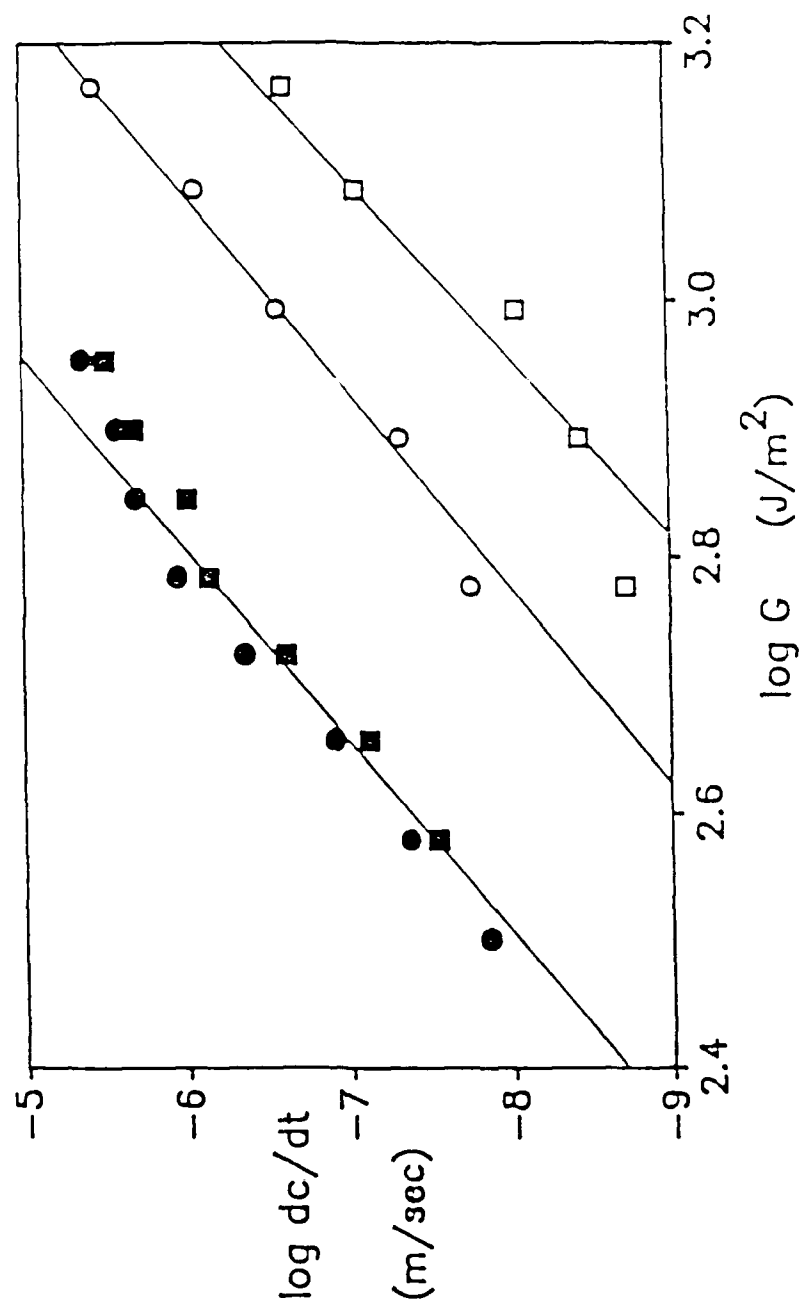FIGURE 9

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FIGURE 10

FIGURE II
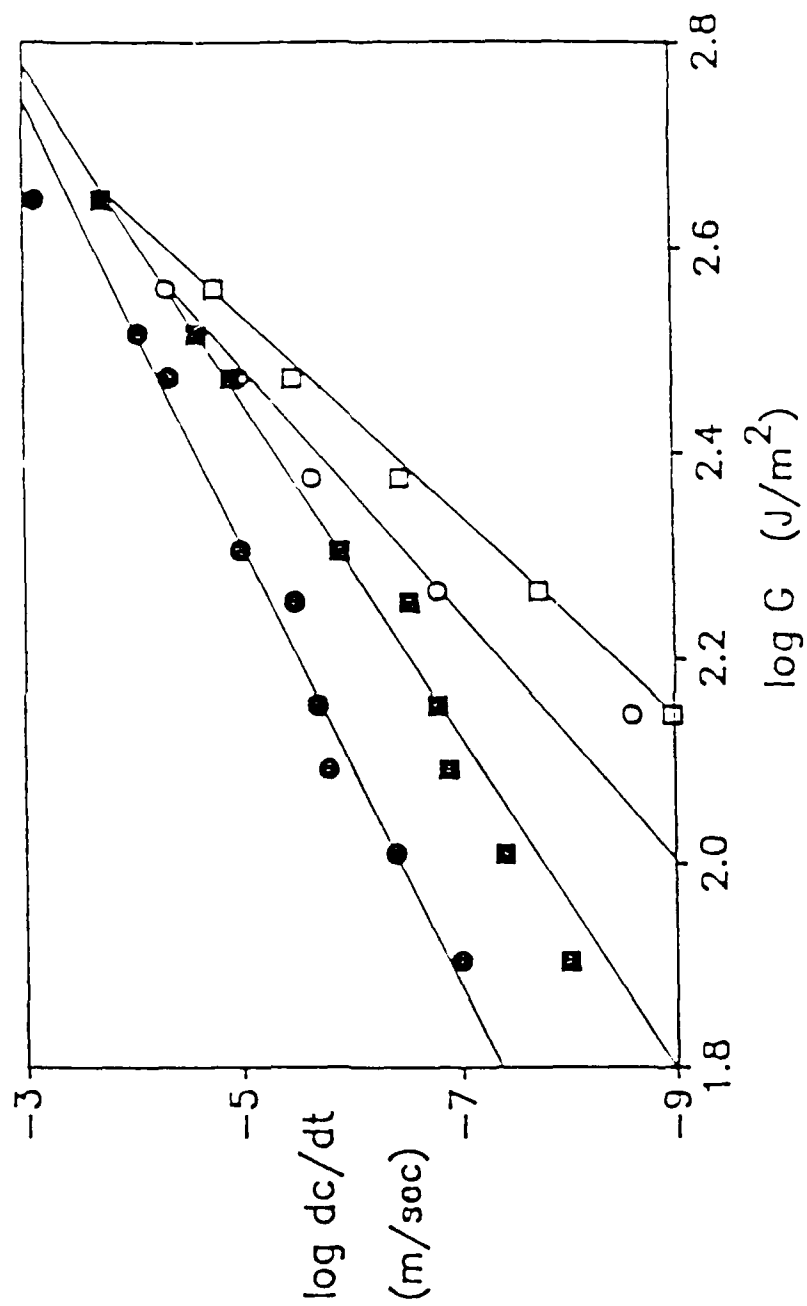
FIGURE 12

FIGURE 13

FIGURE 14

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